Chemical Synthesis: Martin Method

Used to synthesize gold nanoparticles ranging from 1-10nm

Protocol:

Perform all steps in FUME HOOD.

1. Measure 20 mL of distilled-deionized H2O (ddH20) into 50 mL graduated cylinder and pour into 125mL Erlenmeyer flask.
2. Keep some extra ddH2O on ice for later steps in a falcon tube.
3. Weigh out 1.96 mg (0.00196 g) of chloroauric acid trihydrate (HAuCl4*3 H2O) solid on a weigh-boat.

- CAUTION: oxidizes quickly - do not expose to air for longer than required
- Do NOT use metal scoopula - use plastic or wood to transfer the solid
- LIGHT SENSITIVE - use for short duration under light conditions
- Note: chloroauric acid is expensive - try not to be wasteful!

4. Using a micropipet, quickly add ~40 uL of the ddH2O from the flask to the crystals of chloroauric acid trihydrate on the weigh-boat. This should dissolve the solid to generate a yellow mixture on the weigh-boat. Then use the micropipette to transfer the yellow liquid to a 1.5 mL microfuge tube.
   - By avoiding transfer of the solid form of HAuCl4, the possibility of loss of sample is lessened. Transfer as a liquid is preferable.
   - Seal the tube rapidly to limit air exposure of the chemical.
5. Micropipet the HAuCl4 solution directly into the flask and seal the opening with aluminum foil.
6. Add stir bar to flask and mix on plate to allow the solid HAuCl4 to dissolve.
   - Nanoparticle synthesis occurs at this step.
   - Having the flask opening covered with aluminum foil prevents the escape of fumes from the synthesis reaction.
7. Weigh out 1.47 mg (0.00147 g) of trisodium citrate dihydrate solid.
8. Add the trisodium citrate dihydrate to the mixture in the flask with stirring.
9. On ice, prepare 0.1 M sodium borohydride (NaBH4) solution.
   - Pipet 0.65 mL (650 uL) of cold ddH2O into a 1.5mL microfuge tube kept on ice.
   - Weigh out 2.46 mg (0.00246 g) of NaBH4.
   - Put the measured NaBH4 into the microfuge tube containing ddH2O and resuspend by pipetting until the solid is fully dissolved (keep on ice throughout).
10. Add 0.6 mL (600 uL) of the cold 0.1 M NaBH4 solution into the mixture in the Erlenmeyer flask while stirring vigorously on the stir plate. - NOTE: the solution should turn dark red immediately - indicating particle formation.
11. The solution is then tested for presence of nanoparticles using UV spectrophotometry
    - Expected absorbance peak between 510-525 nm.
12. Once nanoparticles are confirmed in solution:
    - Weigh out 0.15 g of L-cysteine solid.
    - Add the L-cysteine to the nanoparticle solution with stirring for ~10 min.
13. Collect nanoparticles by pelleting using centrifugation at 1,000 RPM at room temperature for 50 minutes.
14. Store the solution in 4°C fridge.

Source: Nan, J., Xiao-Yu, Y., Guo-Liang, Y., Ling, S., Jing, L., Wei, G., Ling-Jun, D., *et al*. 2015. “Self-repairing”nanoshell for cell protection. *Chem Sci*, 6: 486-491.

Chemical Synthesis: Turkevich Method

Used to synthesize silver nanoparticles ranging from 15-30nm.

Protocol:

1. Fill a 50 mL falcon tube with ddH2O.
2. Weigh out 3.6 mg (0.0036 g) of AgNO3 powder.
    - Light sensitive - limit light exposure by covering with aluminum foil

3. Measure 20 mL of ddH2O in a 25 mL graduated cylinder. Pour the water into a 100 mL beaker.

4. Add the AgNO3 solid to the ddH2O in the beaker and place a stir bar into the solution to mix it.

5. On the hot plate, stir the solution with the stir bar and boil it to fully dissolve the AgNO3. Place a watch glass on the opening of the beaker to prevent vapours from escaping during boiling.
    - Light sensitive - limit light exposure by covering with aluminum foil
    - During this step, nanoparticle synthesis should occur

6. Measure 4 mL of 1% sodium citrate into a 10 mL graduated cylinder.
    - If the 1% sodium citrate solution is not prepared, 5 mL of sodium citrate solution can be made:

A. Weigh out 0.05 g (50 mg) of sodium citrate solid.

B. Using a 10 mL graduated cylinder, measure 5 mL of ddH2O and pour it into a conical tube.

C. Add the sodium citrate to the conical tube and cap the tube.

Mix the solution by inversion until the solid dissolves.

7. Add the sodium citrate to the AgNO3 solution dropwise into the beaker with continuous stirring.
    - Keep stirring until a yellow-brown color change is observed, at about 10 min - indicating citrate attachment to Ag nanoparticles

8. Remove the solution of citrate-coated nanoparticles from the hot plate and let it cool down at room temperature.

9. Store in 4°C fridge.

Source: Konnova, S. A., Danilushkina, A. A., Fakhrullina, G. I., Akhatova, F. S., Badrutdinov, A. R., & Fakhrullin, R. F. (2015). Silver nanoparticle-coated “cyborg” microorganisms: rapid assembly of polymer-stabilised nanoparticles on microbial cells. *RSC Adv.*, *5*(18), 13530-13537. Retrieved from http://pubs.rsc.org/en/content/articlehtml/2015/ra/c4ra15857a